

# **Feasibility of a Miniature, Low-Voltage Scanning Electron Microscope for In Situ Applications**

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## **Abstract**

The scanning electron microscope is a powerful laboratory analysis tool. However, it has not been exploited as an in situ instrument for applications in Space and elsewhere, principally due to its size. Recent technological developments have enable low-voltage microcolumns to function as a miniature scanning electron microscope (SEM). An attractive feature of the low-voltage operation is the minimization of sample charging on non-conductive, non-prepared samples. A potential drawback, however, of the low-voltage operation is the diminished ability to perform e-beam induced X-ray fluorescence spectroscopy on heavy elements. Proof-of-concept testing with commercial SEMs on "Mars-like" samples has validated the low-voltage SEM as a viable analytical instrument for in situ applications, avoiding the need for sample preparation and demonstrating meaningful X-ray fluorescence spectroscopy with reduced beam voltages.

## Introduction

The scanning electron microscope (SEM) continues to be a powerful analytical tool in the laboratory for a variety of applications. An attractive feature of the SEM is its ability to simultaneously image and perform elemental spectroscopy via e-beam induced X-ray emission. This enables the ability to quantitatively determine the elemental composition of features observed within a sample. The efficient production of *K*-level emission X-rays, typically the strongest signature, usually needs electron beam energies roughly twice that in the expected X-ray line energy.<sup>1</sup> For most commercial SEMs, this requires beam energies of at least 15 or 20 kilovolts (kV). For beam voltages below 3 kV where a miniature flight SEM might operate, the number of elements accessible by induced X-ray fluorescence needs to be examined.

The high beam voltages of a commercial SEM necessitate the use of electromagnetic optics, which continue to be quite large with substantial power requirements. If the beam voltage requirement can be relaxed, a miniature SEM using electrostatic optics with a very small column can be realized. One such device is the microcolumn from Etec Systems, Incorporated.<sup>2,3</sup> The microcolumn uses silicon electrodes that are fabricated with lithographic techniques to form a column length of only 3.5 mm. A Schottky field emission tip is employed to provide a stable, high-brightness source with a small spot size. A small, integrated microchannel plate (MCP) is used as a backscattered electron (BSE) and secondary (SE) electron detector. Operating around 1 kV, the microcolumn has demonstrated 10 nanometer spatial resolution. Other promising miniature column devices are under development elsewhere.<sup>4,5</sup> Efforts are now underway at the Jet Propulsion Laboratory (JPL) to design a miniature flight SEM instrument incorporating miniature electrostatic column technology.

To test the feasibility of a low-voltage SEM operating on samples of interest, several experiments were performed at JPL using two commercial SEMs. For the tests, the Martian Regolith Simulant

JSC Mars-1<sup>4,5</sup> and terrestrial basalt samples were used. The Mars-1 simulant was developed to provide researchers with a candidate "Mars-like" material for use in investigation and instrument development efforts. An analysis of the Mars-1 simulant is contained in the references<sup>6,7</sup> and a derived summary of the volatile-free elemental composition is given in Table I.

### **Low-Voltage Imaging**

An attractive feature of a low-voltage SEM, in addition to the small instrument size, is the ability to image non-conductive, non-prepared samples with a minimum of image artifacts from charging. Usually when imaging most non-conductive samples in the laboratory, a conductive coating is applied. The coating, typically carbon, gold or other evaporable conductor, requires some sophistication in applying and ultimately alters the sample. For applications in Space, for example on the surface of Mars, such coatings would be difficult, at best, to apply robotically and with meaningful reliability. At low voltage near 1 kV, however, the SEM operating conditions (e.g., beam voltage, current, etc.) can be adjusted such that the instrument beam current matches the sample backscattered current, balancing the net charge of the sample.<sup>8</sup>

Using a commercial Hitachi S-400 Scanning Electron Microscope at JPL, low-voltage imaging tests were performed on unprepared Mars-1 simulant. The S-400 SEM was configured with operating parameters that represent a miniature, low-voltage flight SEM. Images were collected at 1 kV beam voltage and approximately 1.4 picoamps (pA) of beam current at TV scan rates. The working distance was set to 5 mm with the sample stage normal to the beam.

Figure 1 shows a SEM image of the Mars-1 simulant at 2500X magnification with 50 nm per pixel resolution (24  $\mu$ m across the FOV). As can be seen, the image is clear with good contrast, exhibiting only a small amount of distortion from charging. Features and textures of the Mars-1 sample are easily discerned. Figure 2 is the same sample observed at 5000X magnification with 12

nm per pixel spatial resolution (6  $\mu\text{m}$  across the FOV). Again the image is clear with good contrast. Some small amount of charging is apparent, due principally to the longer integration times to achieve the higher resolution image. The beam current can simply be reduced to remove these charging effects.

### **Low-Voltage Spectroscopy**

Low-voltage, X-ray emission spectroscopy experiments were performed on both the Mars-1 simulant and terrestrial basalt samples. A Cambridge Stereoscan 250 Mk 2 scanning electron microscope, instrumented with a Kevex 10 mm<sup>2</sup> Si(Li) X-ray detector and integrated multichannel analyzer, was used. The X-ray detector achieves spectral resolution of 109 eV (FWHM) at 677 eV. The detector assembly was positioned 20 mm from the beam target point, corresponding to a subtended solid angle of 0.025 steradians.

X-ray spectra were collected at TV scan rates on all samples. In each case, the sample stage was positioned at 45 degrees to the incident beam at a working distance of 14 mm. All spectra were collected for 1000 seconds (live time) at a beam voltage of 2.5 kV. The beam voltage was selected, as before, to be consistent with the capability of an envisioned flight SEM, such as one incorporating the Etec microcolumn. However, higher voltages are possible in the miniature column technologies with some design modifications.<sup>9</sup>

The Mars-1 spectrum, collected with a beam current of a few pA, is shown in Figure 3. In the case of the basalt, two spectra were collected with beam currents of several tens of pA, one of the entire sample and the other of a specific inclusion feature within the sample (selected because of its obvious visual difference from the rest of the sample). Both basalt spectra are represented in Figure 4. In all spectra, several important elemental lines are clearly resolved. Carbon (277 eV), oxygen (525 eV), aluminum (1487 eV), silicon (1740 eV), and iron (705 eV) are easily identifiable

and quantifiable. Iron, a heavier element, is strongly visible through its *L*-level emission at 705 eV in all samples. Sodium (1041 eV) and magnesium (1254 eV), at single-percent concentrations in the Mars-1 sample, are just identifiable. The observed carbon signature is not from the samples, but from the carbon mounting-pad under each sample. Nitrogen (392 eV), an important biological constituent, was not present, but would be easily observable between the carbon and oxygen peaks.

The data in each spectra were fitted with the spectral fitting program HYPERMET<sup>10</sup> to determine peak centroids, line widths, and total counts of each detected element. The fitted results for the Mars-1 simulant and the basalt samples are given in Table II. As will be noted when reviewing the Mars-1 spectrum, the minor elements, Na and Mg, were not quantified in the fit due to their apparent weakness. For the dominant elements in each sample, the fit results in small uncertainty (few percent) on the total counts. This is encouraging considering the short integration time (1000 s), modest currents (few pA) and small detector solid angle used in these experiments. A miniature flight SEM can be envisioned to be quite sensitive when recognizing that increasing any of these operating parameters is within the expected capability of the miniature column technology. For example, the Etec microcolumn technology can operate with beam currents in excess of 1000 picoamps.<sup>6</sup> Highly integrated X-ray detectors with solid angles approaching 1 steradian for a flight instrument are now under development.

The precision of these low-voltage spectral results is sufficient to begin to resolve and identify substances within samples of interest, collected at Mars and elsewhere. For astrobiologists, the ability to identify carbon, nitrogen and oxygen is a powerful tool in the search for organic material. For geologists, the ability to spectrally quantify the elements, carbon, oxygen and silicon permits the identification of carbonates and silicates. Even though heavier elements, such as, potassium and calcium, are not observable at a beam voltage of 2.5 kV, the other observed elements along with their ratios can provide information for the unique identification of complex mineral types.

## **Conclusion**

These results demonstrate that low-voltage scanning electron microscopes are highly attractive candidates for a miniature flight instrument capable of both fine spatial imaging and elemental (and mineral) identification. In addition to the extremely small column size, a low-voltage SEM has the additional advantage of being able to image "Mars-like", or other non-conductive, samples without the need for complex, sample-altering preparation. Clear, high-resolution imaging of non-prepared, "Mars-like" samples has been demonstrated with minimal charging artifacts. At beam voltages of 2.5 kV, many of the important elemental constituents above the single-percent level in a sample can be readily identified and quantified with short integration times and modest beam currents.

## **Acknowledgments**

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**Table I.** Elemental Mass Fractions of Martian Regolith Simulant JSC Mars-1

Element	Mass Fraction
O	0.4476
Na	0.0178
Mg	0.0205
Al	0.1233
Si	0.2033
P	0.0039
K	0.0050
Ca	0.0443
Ti	0.0228
Mn	0.0023
Fe	0.1091



**Table II.** Elemental Counts from the Mars-1 and Basalt Sample Spectra

Element	Mars-1 Simulant		Entire Basalt Sample		Basalt Inclusion Feature	
	Line Counts	<i>Ratio</i>	Line Counts	<i>Ratio</i>	Line Counts	<i>Ratio</i>
C	34499	0.236	61373	0.125	-	-
O	146054	1	491523	1	292398	1
Fe	24456	0.167	36868	0.075	77171	0.264
Na	-	-	17720	0.036	7494	0.026
Mg	-	-	5395	0.011	13449	0.046
Al	9314	0.064	44881	0.091	9716	0.033
Si	5413	0.037	56275	0.114	28248	0.097

**Figure 1.** SEM Image of Mars-1 Simulant at 1 kV (24  $\mu\text{m}$  FOV)

**Figure 2.** SEM Image of Mars-1 Simulant at 1 kV (6  $\mu\text{m}$  FOV)

**Figure 3.** X-ray Spectrum of Mars-1 Simulant at 2.5 kV

**Figure 4.** X-ray Spectrum of terrestrial basalt sample (shaded) and a feature within the sample (line) at 2.5 kV

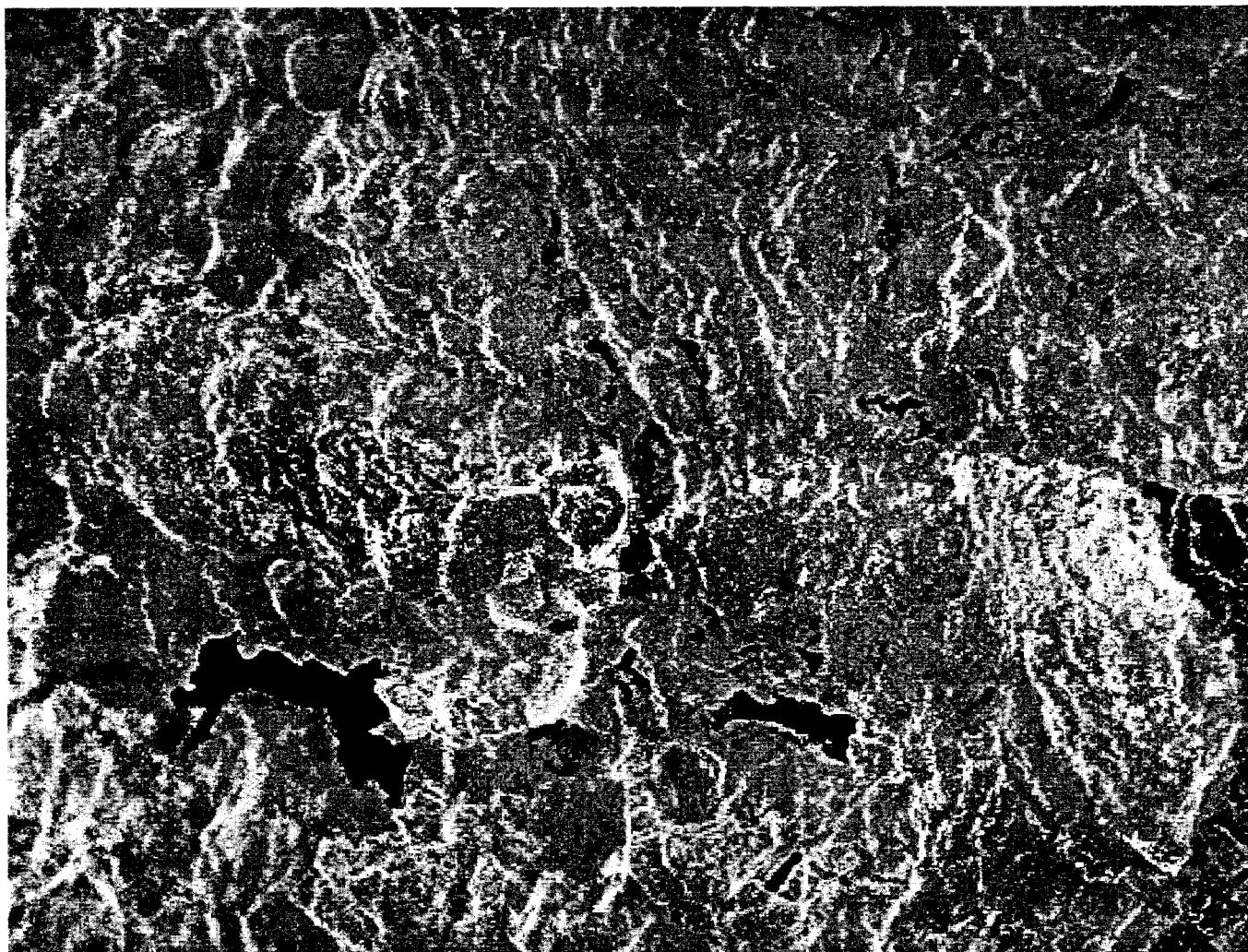


Figure 1, John L. Callas, *Applied Physics Letters*

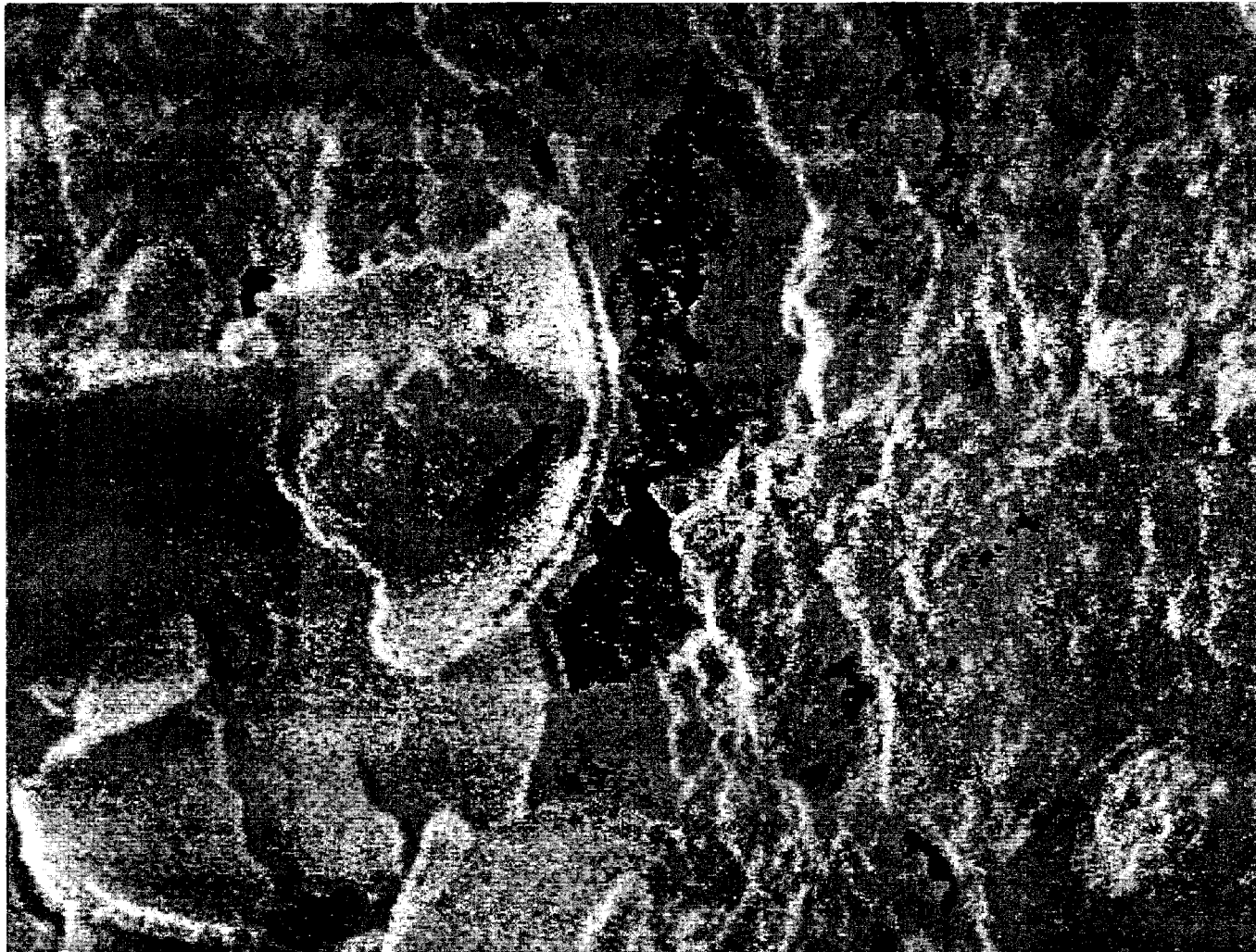


Figure 2, John L. Callas, *Applied Physics Letters*

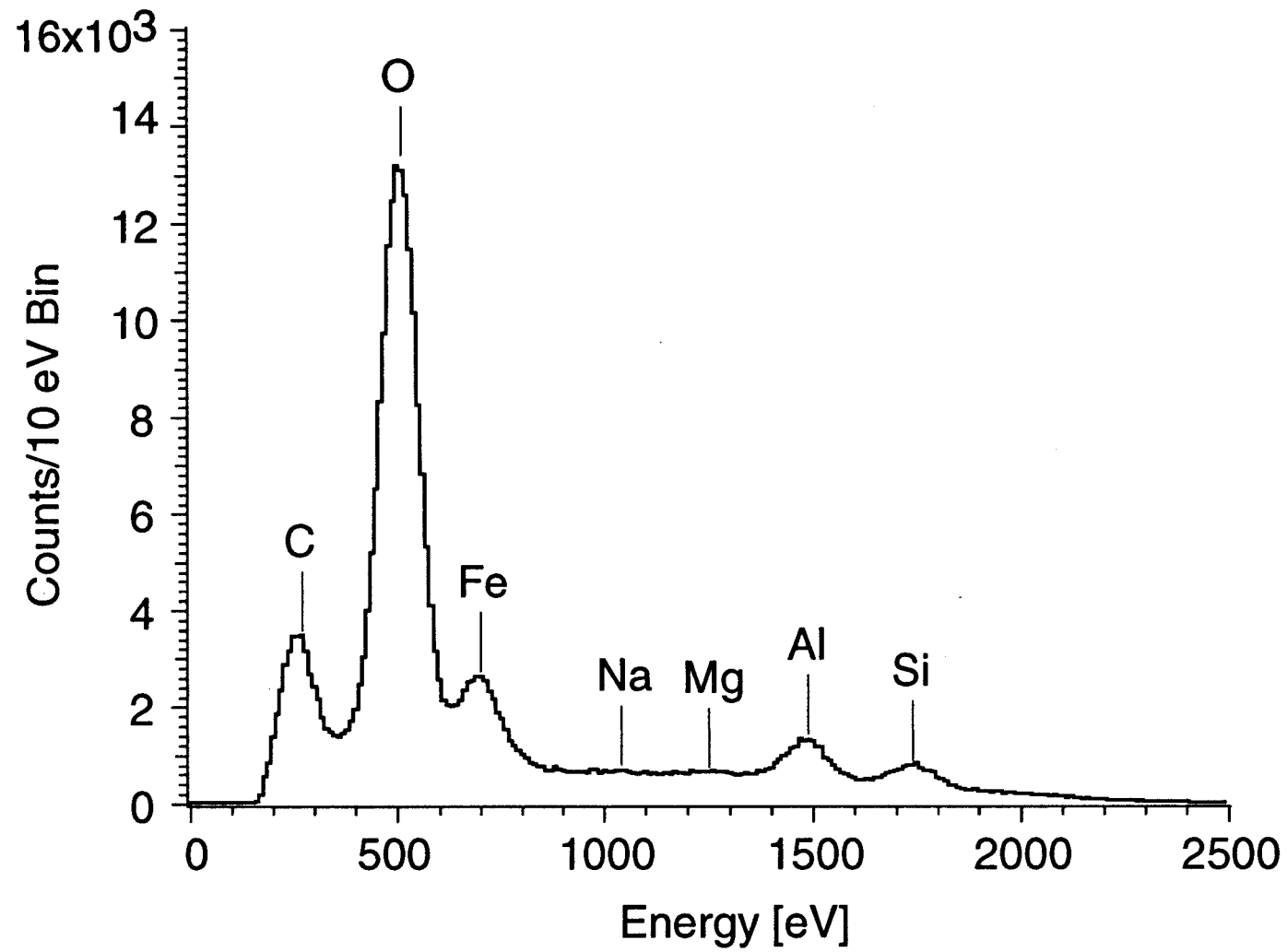


Figure 3, John L. Callas, *Applied Physics Letters*

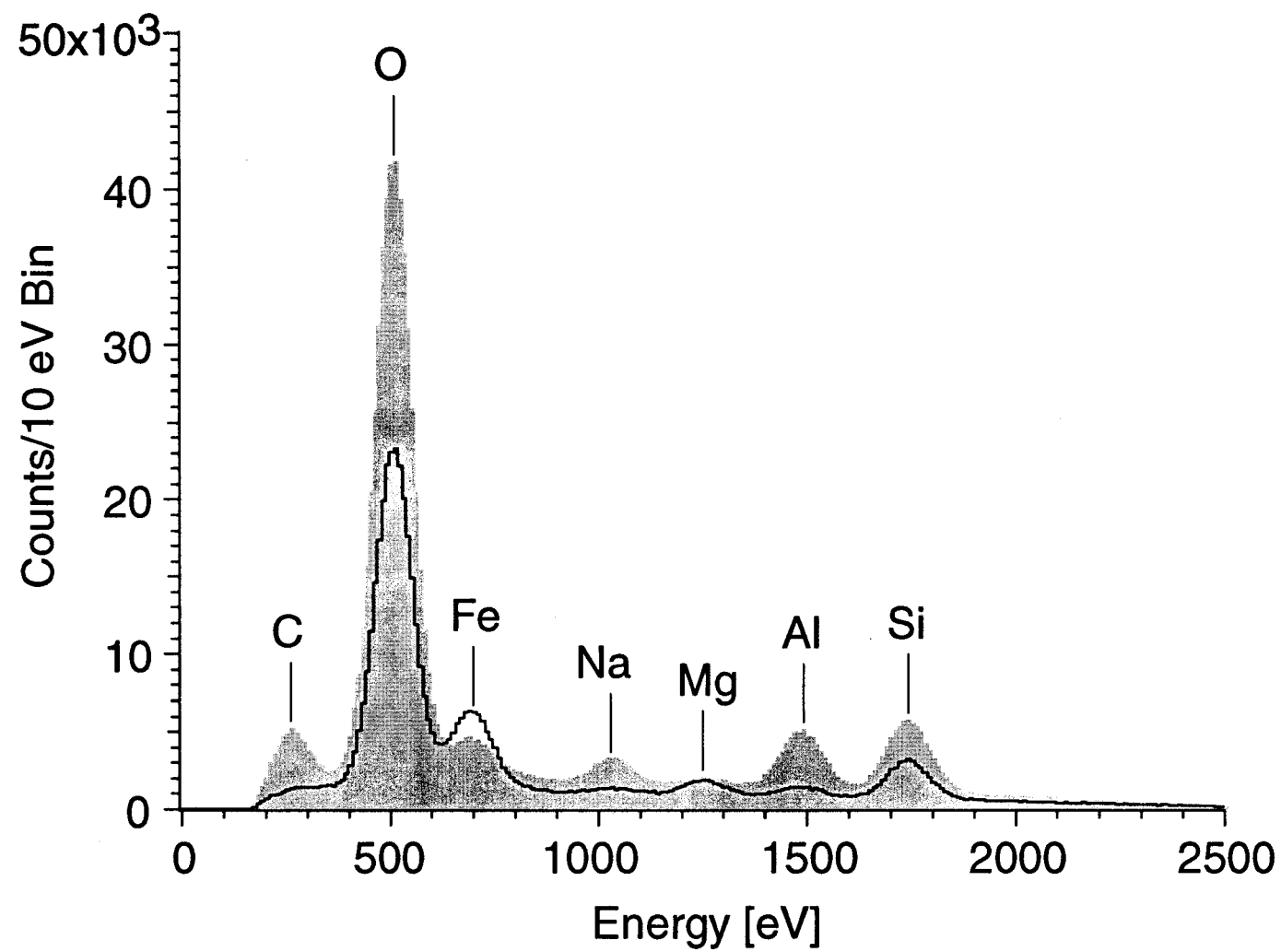


Figure 4, John L. Callas, *Applied Physics Letters*